

contact with a monohydroxy compound or a mixture of said compound and water.

The second invention of the present invention is a method for crystallizing a low molecular weight aromatic polycarbonate characterized in
5 that 100 parts by weight of an uncrystallized low molecular weight aromatic polycarbonate whose main recurrent unit is expressed by the above formula (1), and having an intrinsic viscosity $[\eta]$ of 0.05 to 0.38 and 0.1 to 25 parts by weight of an aromatic monohydroxy compound are subjected to melt mixing, and the obtained mixture is held at a temperature equal to or higher than
10 the glass transition temperature and lower than the melting point of the mixture for crystallizing the low molecular weight aromatic polycarbonate.

The third invention is a method for preparing a high quality polycarbonate resin by heating the low molecular weight aromatic polycarbonate, which has been crystallized by the above method, at a
15 temperature equal to or lower than the melting point of the crystallized product under reduced pressure or in an inert gas flow to convert it into a high polymerization state.

Hereafter the method of the present invention is explained in detail.

"Aromatic polycarbonate" of the present invention is a thermoplastic
20 polymer whose main polymeric recurrent unit is expressed by the above-mentioned formula (1).

The aromatic polycarbonate is a thermoplastic polymer commonly produced by the reaction of an aromatic dihydroxy compound with a carbonate bond-forming compound, and as the aromatic dihydroxy compound
25 used in the production of the polymer, a compound expressed by the following formula (2) is preferably used, and in the formula (2), R_1 , R_2 , R_3 and R_4 , and W are same as those in the above formula (1).

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above-mentioned formula (1) and an intrinsic viscosity $[\eta]$ of 0.05 to 0.38, and 0.1 to 25 parts by weight of an aromatic monohydroxy compound are melt mixed, and the obtained mixture is crystallized by holding it at a temperature equal to or higher than the glass transition temperature, and
5 lower than the melting point of the mixture.

That is, to 100 parts by weight of the low molecular weight aromatic polycarbonate is added 0.1 to 25 parts by weight of an aromatic monohydroxy compound as a plasticizer, and the mixture is melt mixed under the following conditions.

10 Here, preferable examples of the aromatic monohydroxy compound include phenol, benzyl alcohol, phenethyl alcohol, o-chlorophenol, m-chlorophenol, p-chlorophenol, o-cresol, m-cresol, p-cresol, 1-naphthol, 2-naphthol and the like. Among them, phenol, benzyl alcohol, phenethyl alcohol, o-chlorophenol and m-chlorophenol are preferable, and phenol is
15 especially preferable.

The low molecular weight aromatic polycarbonate and an aromatic monohydroxy compound are mixed at a temperature equal to or higher than the glass transition temperature (T_g) of the lower molecular weight aromatic polycarbonate. The melt-mixing temperature is commonly 160 to 250°C,
20 preferably 180 to 220°C.

The method for melt mixing is not specifically limited, as far as a low molecular weight aromatic polycarbonate of a molten state and an aromatic monohydroxy compound can be homogeneously mixed. For example, an aromatic monohydroxy compound may be added to the product at a point
25 when the polymerization reaction of the low molecular weight aromatic polycarbonate has been completed in a vertical type or horizontal type polymerization reactor and mixed, or an aromatic monohydroxy compound may be added in a liquid state to the product by a twin-screw extruder or the like using a plunger pump or the like and melt mixed.

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In the present invention, as mentioned above, a low molecular weight aromatic polycarbonate of a substantially uncrystallized state and an aromatic monohydroxy compound are melt mixed homogeneously at a temperature equal to or higher than the glass transition temperature (T_g) of the low molecular weight aromatic polycarbonate, and subsequently the mixture is crystallized by holding it at a temperature ranging from the glass transition temperature (T_g) to a temperature not exceeding the melting point of the mixture. If the temperature is lower than the glass transition temperature (T_g) of the mixture, the crystallization hardly proceeds, and if it is equal to or higher than the melting point, crystals melt, and objects of the present invention are not achieved. A preferable temperature for the crystallization treatment is a temperature equal to or higher than "the glass transition temperature of the mixture + 20°C", and not exceeding the melting temperature of the mixture, more preferably a temperature equal to or higher than "the glass transition temperature of the mixture + 40°C", and equal to or lower than "the melting point - 5°C".

For example, in the case where an aromatic monohydroxy compound is used as plasticizer for a polycarbonate made of 2,2-bis(4-hydroxyphenyl)propane and diphenyl carbonate, a temperature in the range of about 110 to 220°C is preferred, although it depends the degree of polymerization.

In the present invention, an uncrystallized low molecular weight polycarbonate can be crystallized as mentioned above; however, 0.1 to 25 parts by weight of a crystallized granular aromatic polycarbonate (this is called "crystallized polycarbonate" in the present invention) having substantially identical structure can be added to 100 parts by weight of the low molecular weight polycarbonate and melt mixed at a temperature lower than the melting point of the crystallized aromatic polycarbonate, at need.

In the first method of the present invention, before an uncrystallized low molecular weight polycarbonate is brought into contact with a monohydroxy compound or a mixture of the compound and water, or in the second method, concurrently with the addition of an aromatic monohydroxy compound, the crystallized polycarbonate like this can be melt mixed with the uncrystallized low molecular weight polycarbonate. By this treatment, the crystallization of the uncrystallized low molecular weight polycarbonate can be accelerated.

Here, "substantially identical structure" means that the main recurrent units of the polymers are identical to each other, and that it is no mater to have some differences in the parts having substantially no relation to the main recurrent units, for example, the structures of the terminal groups, the constitution ratio of a terminal group such as hydroxyl group, the degree of branches generated by a side reaction or the like. There is no specific limitation in the intrinsic viscosity $[\eta]$ of the crystallized aromatic polycarbonate, but it is preferably 0.15 to 1.7, more preferably 0.25 to 1.5.

fine particles was not observed at all in this process.

Examples 13

The low molecular weight polycarbonate (oligomer) obtained in the
5 above-mentioned Reference Example 1 was introduced into a twin-screw
extruder. The temperature of the twin-screw extruder was set at 240°C and
the extrusion flow rate was set at 6 g/ml. On the exit side of the twin-screw
extruder, a strand die and an in-liquid chip cutter were placed. The
aperture of the strand die was 2 mm. As the liquid for the in-liquid chip
10 cutter, phenol was used. The temperature of the phenol was set at 60°C.
The oligomer of a molten state extruded from the strand die was cut by the
in-liquid chip cutter into chips. The shapes of the chips of the oligomer
obtained by cutting using the in-liquid chip cutter were cylinders of 2 mm in
diameter and 2 mm in length, and they were homogeneous. After dipped for
15 5 minutes in the liquid of phenol used for the in-liquid chip cutter, the chips
were taken out from the liquid, and dried at 90°C for 60 minutes. The
oligomer obtained after drying had a crystallized state, and this crystallized
product had an intrinsic viscosity $[\eta]$ of 0.15 and a degree of crystallization of
20%.

20 The granular crystallized product obtained above was charged into a
cylindrical reactor having a blowing port for an inert gas at the lower part,
and it was subjected to a solid phase polymerization at 220°C for 10.5 hours
in a nitrogen gas flow at 4 NL/min. The obtained polycarbonate had an
intrinsic viscosity $[\eta]$ of 0.40.

25 The generation of fine particles was not observed in the above
processes.

Reference Example 5

Similarly, 500 parts by weight of the low molecular weight
30 polycarbonate obtained in Reference Example 1 was introduced into an
extruder, the obtained strands were cut into chips by an in-liquid chip cutter,
and the chips were crystallized. As the liquid for the in-liquid chip cutter,
the mixed solution recovered by condensing and liquefying the diphenyl
carbonate and phenol generated as byproducts in the synthetic reaction of

hours. The obtained polycarbonate had an intrinsic viscosity $[\eta]$ of 0.36, a melting point of 279°C and a ΔH of 57 J/g.

Reference Example 6

5 Five hundred parts by weight of the low molecular weight polycarbonate obtained in the above-mentioned Reference Example 1 was mixed with 25 parts by weight of diphenyl carbonate, the mixture was melt extruded from a twin-screw extruder at 220°C, the extruded matter was taken out on a hot plate of 165°C, it was held there for 20 minutes for
10 crystallization, and a crystallized product having particle diameters of 0.6 to 1.4 mm was obtained. The obtained crystallized product had an intrinsic viscosity $[\eta]$ of 0.15, a melting point of 216°C, a ΔH of 25 J/g and a degree of crystallization of 30%.

 The polycarbonate was charged into a polymerization apparatus
15 provided with a stirrer and a vacuum distillation system, and a solid phase polymerization reaction was carried out under a reduced pressure of 0.15 mmHg while slowly stirring at 180°C for 2 hours, subsequently for 5 hours after temperature elevation to 220°C, and further for 15 hours at 230°C. The polycarbonate thus obtained had an intrinsic viscosity $[\eta]$ of 0.36, a melting
20 point of 273°C and a ΔH of 45 J/g.

Reference Example 7

 A mixture consisting of 500 parts by weight of the low molecular weight polycarbonate obtained in the above-mentioned Reference Example 1,
25 25 parts by weight of diphenyl polycarbonate and 25 parts by weight of the crystallized polycarbonate obtained in Reference Example 2 was melt extruded from a twin-screw extruder at 220°C, the extruded matter was taken out on a hot plate of 165°C, it was held there for 15 minutes for crystallization, and a crystallized product having particle diameters of 0.6 to
30 1.4 mm was obtained. The obtained crystallized product had an intrinsic viscosity $[\eta]$ of 0.14, a melting point of 219.4°C, a ΔH of 22.8 J/g and a degree of crystallization of 29%.

 By using the polycarbonate, solid phase polymerization was carried out in the same way as in Example 2, resulting in the preparation of an